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13. ABSTRACT (Maximum 200 words) This program intended to provide substantial improvements to conditions that affect imaging nanoscale structures with atomic resolution and chemical specificity by magnetic resonance force microscopy. This is a final report of an 18 months effort. During this period we the following activities: We increased the sensitivity of the cantilever displacement readout and therefore the force sensitivity achievable with mechanical detection. We implemented an approach to reduce the spin noise in MRFM by shifting to higher frequencies the higher order cantilever vibrational modes. We build a low temperature microscope that operates in vacuum at 4K in a field of up to 9Tesla and tested the system. Finally, we selected CaF ₂ as the first sample to test the feasibility of performing magnetic resonance force spectroscopy within the resonance slice, prepared samples and characterized the sample.				
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1. Foreword

We conducted an eighteen months research program in support of the efforts to develop a non-destructive direct observation technique capable of manipulating and producing three-dimensional images of mesoscale materials and structures in their natural environments with chemical specificity and atomic resolution. Our goal was to improve the force detection technology in order to increase the resolution of magnetic resonance force microscopy (MRFM).

This program provided improvements to the conditions that affect the realization of atomic resolution imaging of nanoscale structures with chemical specificity and atomic resolution. It centers on two aspects that affect the resolution of MRFM.

These two aspects were:

- a) The cantilever displacement readout and therefore the force sensitivity achievable with mechanical detection when thermal noise is no longer the dominant source.
- b) The spin noise produced by the unwanted motion of the field gradient generating magnetic particle by higher cantilever oscillation modes.

2. Statement of the problem studied

Force detection in MRFM instruments is realized by measuring the deflection of a floppy cantilever. Brownian motion is the biggest contribution to the noise in MRFM. By lowering the temperature of the mechanical oscillator its random fluctuations can be reduced. MRFM experiments operating at fractions of one Kelvin temperatures are in progress. After Brownian motion, the next contribution to the noise in mechanical detection of magnetic resonance comes from the process of detecting cantilever displacement. Cantilever displacement sensing or readout noise affects MRFM measurements. In addition to readout noise, spin noise is present in MRFM measurements due to unwanted motion of the field gradient generating magnetic particle by higher cantilever oscillation modes.

In this work we addressed cantilever displacement readout noise and spin noise problems.

Cantilever displacements have been typically detected by optical techniques either by beam deflection or optical interferometry, with the highest displacement resolution so far obtained by interferometry. Cantilever displacement noise floor levels in the range of $10^{-4} \text{Å}/\sqrt{\text{Hz}}$ have been reported.

Our objective in this work was to increase the resolution of interferometric detection in order to bring the noise floor levels closer to their fundamental quantum limit (the noise spectral density associated with fluctuations in optical power due to light quantization and the noise limit of the photo detector). In addition, to provide a cantilever design that enhances the fundamental cantilever oscillation mode and pushes the high order harmonics to frequencies outside of the bandwidth of relevance to the MRFM experiments.

3. Summary of the most important results

a. Modeling and optimization of interferometric displacement detection

An important question in interferometric force detection schemes such as Magnetic Resonance Force Microscopy (MRFM)[1] or Atomic Force Microscopy (AFM)[1-3], is how to improve the detection sensitivity. Through a detailed optical analysis and optimization of the fiber optic interferometer. Such improvements were relevant in the efforts toward single spin detection and sub-micron resolution imaging in MRFM[4] and highly sensitive noncontact AFM[2, 3, 5].

We used a plane wave propagation model that takes into account the complex index of refraction and extinction coefficient of the various components of interferometric force detection schemes. The model enabled us to enhance the interferometer finesse, subsequently improving the cantilever displacement detection signal to noise ratio (SNR). Using this model we obtained optimal cantilever geometries for minimizing deleterious heating effects due to light absorption while maximizing reflection.

The majority of fiber optic interferometer detection schemes for AFM and MRFM rely on a design published by D. Rugar et. al. in 1989[6]. In this technique, a cleaved optical fiber is placed in close proximity to a micro-mechanical cantilever. The light reentering the fiber from reflections off the cantilever and the fiber-air interface produces fringes as a function of the wavelength of the light and distance between the cantilever and the end of the fiber. Detection of the force on the cantilever is made by measuring the cantilever's deflection with its inherent displacement amplification gain provided by the ratio of the quality factor, Q , to the cantilever spring constant, k , when operating at the cantilever resonance frequency. This optical arrangement forms a simple Fabry-Perot interferometer. With a typical 4% reflection from a cleaved fiber-air interface and the reflection of either a Au-coated SiN or Si cantilever the Fabry-Perot fiber optic interferometer achieves a typical finesse of $\mathcal{F} \leq 1$. This method has been employed by members of the community performing force detected measurements, yet no rigorous analysis of the interferometric system and its components has been given. The model developed in our work highlights how the experimenter can enhance the finesse of the interferometer in such detection schemes by increasing the overall reflectance of the components comprising the cavity either with a metallic layer or a dielectric reflector, subsequently improving the SNR. An important consequence of this analysis showed that too thick a reflective coating on the fiber is detrimental to the SNR.

b. Minimization of cantilever heating

The drive to improve sensitivity in a number of cantilever-based force measurements has pushed the development of low damping (high Q), soft (low k) silicon cantilevers. In force-detected scanned probe microscopy techniques employing cantilevers, the thermo-mechanical motion is a major contributor to the noise spectral density. The minimum detectable force for a cantilever, F_{min} , on the basis of the thermomechanical noise, is expressed as $F_{min} = (4k_B T B \Gamma)^{1/2}$ where $k_B T$ is the thermal energy, $\Gamma = k/Q$ is the mechanical resistance, and B is the bandwidth. The main method to reduce F_{min} has involved operating at 4K[7] or in the milliKelvin[8] regime. At these very low temperatures, the small heating effects of the incident laser light become apparent and it behooves the experimenter to consider minimizing such effects. We use our model to analyze the absorption and reflection properties for silicon cantilevers, so that deleterious heating effects caused by the incident laser light may be minimized.

Asheghi and coworkers[9] report that the thermal conductivity for thin Si layers and devices as a function of temperature below 100 K decreases by as much as a factor of 20 from crystalline Si. The thermal conductivity for crystalline Si, from which most cantilevers are manufactured, decreases from a peak of 5000 W/(m•K) at 20 K to less than 7 W/(m•K) below 1 K[10]. At 4 K, the thermal conductivity of the Si

cantilever is $\sim 300 \text{ W}/(\text{m}\cdot\text{K})$ and therefore the rise in temperature per light power absorption corresponds to $1 \text{ mK}/10 \text{ fW}$. We minimized the light absorption by employing radiation below the Si band gap, 1.1eV . Under these conditions the absorption coefficient drops below 10^{-6} and the cantilever becomes essentially nonabsorbing. In order to increase the reflectance of a Si cantilever while minimizing the absorption, the cantilever thickness, t_c , was made such that $t_c = m\lambda/4n$, where n is the index of refraction of the cantilever material, λ is the wavelength and m is a positive integer.

We applied our model to calculate the extent of light absorption and reflection as a function of silicon thickness for various wavelengths of commercially available light sources. As the extent of light absorption and reflection on silicon cantilevers does not depend on the reflective film placed on the fiber, we ignored this and assumed an Air/Silicon/Air interface. The reflectance, absorbance and ratio of reflection to absorption (as a measure of the efficiency) of a silicon cantilever with various wavelengths of were calculated. For our calculations, we used the optical constants of bulk materials compiled by Palik[11].

We obtained an optimal thickness and wavelength conditions to use for the cantilever. The absorption decreases at wavelengths longer than the 1.1eV Si band gap. The maximum reflection of a cantilever was achieved for an optical thickness $nt = m(\lambda/4)$ where $m = 1, 2, \dots$ and for $\lambda = 1.5\mu\text{m}$ the optimal thickness for a Si cantilever at the illumination spot is 1100\AA .

c. Interferometer optimization

We used our model to predict the effects of placing a thin metallic reflector on the surface of the fiber in order to improve the interferometer finesse. The cantilever displacement, Δz , was obtained by approximating the detector response, R , around the maximum slope with a linear function of the cantilever displacement.

$$\frac{\Delta R}{\Delta z} = \frac{2\pi}{\lambda} \frac{\partial P_r}{\partial \delta_r} \bigg|_{\max}$$

Given that the signal is proportional to the light intensity, to a first approximation the SNR for the interferometric detection is proportional to the finesse, \mathcal{F} .

The finesse of the Fabry-Pérot fiber-optic interferometer is a function of the Fabry-Pérot cavity reflectance and by enhancing the reflectance of the cavity the displacement resolution was increased.

In order to increase the finesse of the interferometer we added a reflective coating to the fiber/air interface. We used the plane wave propagation model that we developed to predict the effects of a semitransparent coating. The results of the calculation for an optimized thickness Si cantilever (thickness= 1100\AA) at $\lambda=1.5\mu\text{m}$ point to an optimal value of the reflective coating. The reflectance of the fiber as a function of a film thickness for gold, silver, and aluminum were calculated. We decided to apply a gold film to the fiber, as this has a higher reflectance than aluminum at $1.5\mu\text{m}$, and is more stable than silver under ambient conditions. The calculation showed that up to a factor of 16 in the slope, $\Delta R/\Delta z$, can be achieved by increasing the finesse of the interferometer with the application of a reflective gold coating at the end of the fiber. An important result of the calculation is to note that too thick a coating is in fact detrimental.

d. Experimental confirmation of cantilever displacement resolution enhancement

To experimentally achieve the required thickness of metallic coating on the fiber, we designed a special vapor deposition chamber with a fiber optic feed-through that allows us to monitor the reflectance on the end of the fiber while performing the deposition. With a slow rate of evaporation of gold in this chamber,

$\sim 7 \text{ \AA/s}$, we were able to achieve good film deposition control. The application of a gold film of 100 \AA brought the reflectance from a $R = 0.04$ value to $R = 0.68$.

We verified the expected signal enhancement of a 68% reflectance Au coated fiber compared to a 4% reflectance cleaved fiber, by measuring the Brownian motion of a commercially available triangular AFM cantilever $180 \mu\text{m}$ in length. Our intention for using an AFM cantilever, rather than a larger reflecting surface, was to also verify the ease of use of a gold-coated fiber in a real experimental situation applicable in force detection measurements. There was concern as to the ease of aligning the gold-coated fiber correctly, so as to observe the expected improved finesse. We found that with relative ease, the fiber could be aligned with respect to the cantilever to produce the expected signal response. These fibers were integrated into our experimental apparatus for magnetic resonance force microscopy experiments.

e. Cantilever Optimization

We developed a cantilever design that maximizes reflectance while minimizing heating without compromising other parameters of the cantilever. In collaboration with Professor Sy-Hwang Liou from the University of Nebraska at Lincoln who used the focused ion beam at his facility to carve a $\lambda/4n$ thick palette (where n is the index of refraction of Si at $\lambda = 1.5 \mu\text{m}$) at the end of a Si cantilever. This cantilever had a $\sim 60\%$ reflectance at $\lambda = 1.5 \mu\text{m}$.

f. Detector Optimization

We designed and build a passive quenching circuit to operate the InGaAs APD in Geiger mode. The circuit worked well and provided us counting rates up to 5MHz which correspond to a dead time of a $\sim 150 \text{ ns}$. One problem we did not anticipate is the extent of the number of dark counts when operating in Geiger mode for this device. The APD in Geiger mode just above the bias threshold had $\geq 150,000$ counts/second, even upon cooling the APD never got below 100,000 counts/second. This meant that this InGaAs device which was grown on top of InP had a lot of electron traps produced by defects at the InGaAs-InP interface. The large number of traps made using these devices with quenching approach unsuitable for our application. We would have to gate them which would mean a substantial increase in the complexity of the electronics with yet unconfirmed benefits. We know that there are new APD's with better epitaxy that should come in to the market in the next year. We therefore decided to wait. In the mean time we reverted to using the InGaAs PIN photodiodes.

g. Reduction of cantilever generated spin noise

D. Rugar group reported[12] that higher order modes of a cantilever can couple to the sample spin system and increase spin de-coherence (add spin noise into the system). Rugar's group designed and implemented a new cantilever fabrication process to mass-load the cantilevers in order to shift to higher frequencies the vibration of the higher order modes. We obtained a similar effect by keeping the cantilever as rigid as possible and make it bend at a hinge next to the base. In addition to carving the optical palette (as reported above), we used a focused ion beam to carve a hinge at the base of the cantilever in order to reduce the spring constant (the original resonance frequency of this cantilever was 60kHz) and push the response of the higher order bending modes to higher frequencies.

h. Design and construction of a low temperature high field MRFM apparatus

We designed and built a low temperature MRFM to operate at 4 K in a super-insulated Dewar under a maximum field of 9 Tesla. Modifications in the facilities were made in order to accommodate the dimensions of the Dewar that holds the 9 Tesla 2.5" bore magnet. A 4 feet deep pit and a 1/2 Ton lifting system with an I beam mounted trolley was build in the laboratory to house the cryostat and the magnet.

Temperature compensation features were incorporated into the design in order to facilitate the low temperature operation and alignment of the various components. The microscope consists of three plates two stacked in sequence by rods, springs and ball bearings. The two outer plates were made out of brass and the central plate is made out of oxygen free copper. The copper plate in the center contains the MRFM cantilever with an oriented magnetic particle mounted onto it. The upper plate holds the optical fiber that monitors the cantilever displacement. The lower plate holds two concentric piezoelectric ceramics that hold an RF coil and the sample and make it possible to scan the sample with respect to the cantilever. The outer plates have translation and tilt capabilities with respect to the copper plate at the center in order to align the fiber and the sample with respect to the cantilever. Alignment was performed at room temperature with the idea that the relative changes in the relative position of the different components upon cool down would be minimal or small at worst. A rapid cool down was achieved by incorporating a cold finger inside the vacuum chamber. Heat was driven away from the microscope through copper braid soldered between the microscope and the cold finger. Vibration isolation was achieved by a three soft spring suspension. Other than scanning the sample by the action of the piezoelectric ceramics, all adjustments are made a room temperature and ambient. We implemented an external cavity wavelength tuning that allows us to select the laser wavelength so that the sample to cantilever distance occurs at the position of maximum sensitivity.

i. Experimental testing of the MRFM design

In our first attempts we used bare Si_3N_4 cantilevers to which we deposited a layer of Au at the end and we attached and oriented a Sm-Co particle. Under the sample we wrapped a 10 turn coil that allows us to drive the cantilever. Test showed that 4K equilibrium under high vacuum conditions was achieved in a period of 90 minutes. Several cool down tests were performed on the system an initial misalignment upon cool down problem was initially discovered and subsequently corrected. Periodic excitation of the cantilever and enhancements in force detection as described in previous sections were confirmed.

j. Preparation and characterization of samples to test the feasibility of performing magnetic resonance force spectroscopy within the resonance slice.

After demonstrating the improvements in force sensitivity that we proposed we started designing and preparing for experiments for the second phase of the DARPA MOSAIC program. In this phase we were planning to perform magnetic resonance force spectroscopy within the confines of a resonance slice in the presence of field gradients up to 5 orders of magnitude higher than ever attempted.

Demonstrating experimental control of the dipolar Hamiltonian in the presence of a large gradient field, remains an open question in MRFM. If successful, these methods would allow for well-known multiple-quantum NMR spectroscopy to be performed over a spatial dimension of the resonance slice in the sample (can be as small as a few nanometers). We intend to modulate the internal dynamics by a technique known as Average Hamiltonian Theory (developed by J. S. Waugh and Coworkers)[13]. This is a well established technique in Magnetic Resonance that has significantly impacted both solid state NMR imaging and solid state NMR spectroscopy. In the process we intend to apply a well-defined periodic sequence of RF pulses that induces an experimentally controllable time dependence on the otherwise constant internal Hamiltonians (for instance gradient, dipolar and chemical shift Hamiltonians, but may also include RF pulse error Hamiltonians as well). The time evolution (governed by the Hamiltonian) of the system will be experimentally controllable by the choice of the RF pulse sequence. Our first experiment was to perform a Magic Echo sequence in a doped Ce^{3+} sample of CaF_2 . We believe this to be a necessary step before attempting any type of magnetic resonance force spectroscopy. Our system of choice is single crystal of 1wt.% Ce^{3+} doped sample of CaF_2 . We picked CaF_2 because for this system the full internal Hamiltonian is well defined; it is just the dipolar coupling (the system is ~ 300 degrees below the Debye Temperature at room temperature and is therefore well-separated from the lattice). Single crystals of CaF_2 have very large T_1 and $T_{1\rho}$ at low temperatures (several minutes long). In order to avoid having such large relaxation times

we selected samples of CaF_2 doped with 1 wt.% Ce^{3+} . We characterized our samples of CaF_2 and obtained measured values of T_1 at room temperature from a sample we send to the laboratory of Professor David Cory at MIT. The results revealed a sample with a $T_1 = 82\text{ms}$ at room temperature. Based on relaxation studies performed by Humphries and Day[14] on similar samples we estimate that at 4K $T_1 \sim 100\text{ ms}$ and $T_{1\rho} \sim 10\text{ms}$.

At this point the DARPA MOSAIC program was cancelled and we ran out of funds to continue pursuing this endeavor.

4. List of publications, presentations and technical reports

a) Manuscripts submitted for publication

“Analysis and optimization of interferometric displacement detection in force detected measurements”
Raúl Fainchtein, Gregory S. Boutis and Terry E. Phillips, manuscript submitted for publication to the Journal of Applied Physics.

b) Presentations at technical meetings but not published in conference proceedings

“Improvement of Mechanical Detection of Magnetic Resonance”, Raul Fainchtein, DARPA MOSAIC Kickoff Meeting. Washington, D.C. June 3-4, 2002.

“Improvement of Mechanical Detection of Magnetic Resonance”, Raul Fainchtein, 1st Review of the DARPA MOSAIC Program, Review and Traveling Road Show, Los Angeles, CA, October 16-18, 2002.

“Improvement of Mechanical Detection of Magnetic Resonance”, Raul Fainchtein, March Meeting of the American Physical Society, Austin, TX, March 3-7, 2003.

“Improvement of Mechanical Detection of Magnetic Resonance”, Raul Fainchtein and Gregory S. Boutis, 2nd Review of the DARPA MOSAIC Program, Review and Traveling Road Show, Seattle, WA, April 29-30, 2003

“Improvement of Mechanical Detection of Magnetic Resonance”, Raul Fainchtein, 3rd Review of the DARPA MOSAIC Program, Arlington, VA, October 22, 2003.

Low Temperature Magnetic Resonance Force Spectroscopy and Imaging, Raul Fainchtein, Gregory S. Boutis, Terry E. Phillips (Johns Hopkins University Applied Physics Laboratory, Laurel, Maryland 20723-6099), March Meeting of the American Physical Society, Montreal, Quebec, Canada, March 22-26, 2004.

Multiple Pulse Applications in Magnetic Resonance Force Spectroscopy, Gregory S. Boutis, Terry E. Phillips, Raul Fainchtein, (Johns Hopkins University Applied Physics Laboratory, Laurel, Maryland 20723-6099), March Meeting of the American Physical Society, Montreal, Quebec, Canada, March 22-26, 2004.

c) Technical reports submitted to ARO

First end of the year interim report covering a period from June 3, 2002 to December 31, 2002.

Second end of the year interim report covering a period from January 1, 2003 to December 31, 2003.

5. List of participating scientific personnel

Raúl Fainchtein Ph.D.

Principal Investigator

Dr. Fainchtein is a member of the technical staff at the Johns Hopkins University Applied Physics Laboratory. He managed the project and directed all scientific and technical aspects of the work. In addition Dr Fainchtein performed some of the measurements and calculations and was the advisor of Dr. Boutis; a postdoctoral fellow working in this project.

Robert Osiander, Ph.D.

Collaborator

Dr. Osiander is a member of the technical staff at the Johns Hopkins University Applied Physics Laboratory. He was in charge of the custom cantilever fabrication. Dr. Osiander was assigned to other duties by the institution and left the project 6 months after its start.

Terry E. Phillips, Ph.D.

Investigator

Dr. Phillips is a member of the technical staff at the Johns Hopkins University Applied Physics Laboratory. He joined the project and was in charge of the design and construction of the specialized electronics needed for the project.

Gregory S. Boutis, Ph.D.

Postdoctoral Fellow

Dr. Boutis joined the project as a postdoctoral fellow in February of 2003 through a fellowship managed by the American Society for Engineering Education. He completed his training appointment on January 31, 2004. Dr. Boutis performed a lot of the technical aspects of this work. He is currently at Northrup Grumman and has been offered a faculty appointment at The City university of New York.

Sy-Hwang Liou, Ph.D.

Collaborator

Dr. Liou is a professor of physics at University of Nebraska at Lincoln. He modified for us some commercial Si and SiN cantilevers. He used a focused ion beam at his facility to modify the shape and coating of some commercial cantilevers.

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